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Final Progress Report

Design and In-Situ Processing of Metal-Ceramic and Ceramic-Ceramic Microstructures

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13. ABSTRACT *(Maximum 200 words)*
In-situ composites comprising phases of the Ni-A1-O systems are selected for investigation for high temperature applications. Metal-ceramic microstructures have been synthesized in situ by a variety of novel processing techniques, including the partial reduction of oxide compounds and displacement reactions and sol-gel processing for example, the PI has formed Ni-A1 O microstructures in situ by partial reduction of NiA1 O at 1100 C. Equipment and experimental procedures have been developed for performing strength, fracture toughness, and creep experiments on small samples of metal-ceramic composites at room and elevated temperatures. Mechanical properties measurements and microstructural studies have been made with the goal of developing an understanding of the microstructural features of composites that are desirable for good mechanical properties. Micromechanical calculations are being made to develop basic rules and criteria for optimizing the mechanical behavior of the in situ processed metal-ceramic composites operating under high temperature conditions. Analytical and numerical models are being developed to simulate the deformation and calculate their statistical strength and fracture toughness properties.

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I. Objectives

An AFOSR-URI Program for the Design and Synthesis of Advanced Materials was established at Cornell University in May 1993 to bring together faculty working in materials science, chemistry and mechanics. The funding from the AFOSR was used primarily to support the research of graduate students working on advanced materials projects and develop specialized research facilities critical for these projects, including a Processing Laboratory, a Mechanical Testing Laboratory and two computational workstations. The faculty supported by the Program includes Dieckmann, Giannelis and Sass in the Department of Materials Science and Engineering; Hui, Phoenix and Zehnder in the Department of Theoretical and Applied Mechanics; and Burlitch in the Department of Chemistry. This program has officially ended April 30, 1997.

The philosophy of the Program was to produce metal-ceramic microstructures by a variety of innovative *in situ* processing techniques; then to test them to evaluate their mechanical properties for correlation with microstructures as a means of developing optimum properties. Mechanics modelling is performed to calculate the mechanical properties associated with the microstructures. Finally, after checking the validity of the predictions of the mechanics calculations by comparison with the experimental measurements, the theoretical models are used to predict microstructures (e.g. size, morphology and distribution of the metallic and ceramic phases) that give optimum mechanical properties. These desirable microstructures are then to be synthesized.

II. Status of the Effort

In-situ composites have the potential to be used as materials in demanding high temperature applications. For the production of such materials it is very important to understand the reactions that can be used for producing in-situ composites. Because of weight and oxidation resistance consideration in-situ composites comprising phases of the Ni-Al-O systems are very promising for high temperature applications. Metal-ceramic microstructures have been synthesized *in situ* by a variety of novel processing techniques, including the partial reduction of oxide compounds, displacement reactions and sol-gel processing, with the goal to better understand the kinetics of such reactions, the mechanisms of such processes and all factors that are important for the microstructure development. Equipment and experimental procedures have been developed for performing strength, fracture toughness, and creep experiments on small samples of metal-ceramic composites at room and elevated temperatures. Mechanical properties measurements and microstructural studies have been made with the goal of developing an understanding of the microstructural features of composites that are desirable for good mechanical properties. Micromechanical calculations are being made to develop basic rules and criteria for optimizing the mechanical behavior of the *in situ* processed metal-ceramic composites operating under high

temperature conditions. Analytical and numerical models are being developed to simulate the deformation and calculate their statistical strength and fracture toughness properties.

III. Accomplishments/New Findings

A. *In Situ* Processing

Sass has formed Ni-Al_2O_3 microstructures*in situ* by partial reduction of $NiAl_2O_4$ at 1100°C and 1300°C. The influence of TiO_2-doping on the kinetics of the partial reduction reaction and the microstructure and properties of the composite microstructures obtained after partial reduction was examined. At 1100°C, the Ni and α-Al_2O_3 phases formed more rapidly in the presence than the absence of TiO_2-doping. Many small pores were present in the a-Al_2O_3 grains. At 1300°C, TiO_2-doping changed the kinetics of the reduction reaction from interfacial reaction-controlled to diffusion-controlled. Elongated Ni and α-Al_2O_3 grains were present close to the reduction front and equiaxed Ni and α-Al_2O_3 grains formed far from the reduction front. Ti segregation was found at both nickel-alumina interfaces and alumina grain boundaries. The amount of porosity in the reduced microstructures decreased in the presence of TiO_2 doping, which resulted in a higher Young's modulus for the reduced microstructures. The fracture toughnesses of the Ni-Al_2O_3 composites obtained by partial reduction of TiO_2-doped $NiAl_2O_4$ samples were lower than those of the undoped samples. The role of TiO_2 in controlling the kinetics of the reduction reaction and the microstructure and properties of the resultant Ni-Al_2O_3 composites was elucidated.

In a previous report, Dieckmann discussed the formation of alternating Al_2O_3 and Ni_2Al_3 layers parallel to the product band/NiO interface by reacting NiO with Ni_2Al_3 in equilibrium with a Ni-Al alloy at 1000°C. While such a morphology is known to be formed in other solid state reactions, to our knowledge the formation of a periodic layer structure comprising an oxide and an intermetallic phase in a displacement reaction has never been observed before. To determine the generality of a Liesegang-like phenomenon consisting of these types of phases, Dieckmann extended displacement reaction studies to other systems of the type Al-Me-O, where the metallic reactant is an aluminum-rich intermetallic phase and the oxide reactant a transition metal oxide, Me_xO_y.

In the Co-Al-O system, the reactants were CoO and an intermetallic reactant whose starting material contained approximately 19 at% Co. At the reaction temperature of 1000°C, this intermetallic reactant is expected to be composed of Co_4Al_{13} and 12 at% Co-Al liquid alloy. The observed microstructure after 4 hours at 1000°C is shown in Figure 1. The reaction product shown in this figure consists of periodic layers of an oxide and an intermetallic phase. The alternating layers are Co_4Al_{13} (gray) and Al_2O_3 (black). In addition, a layer of metallic cobalt (very bright) was found at the interface between CoO and the periodic layer product zone. In the Fe-Al-O

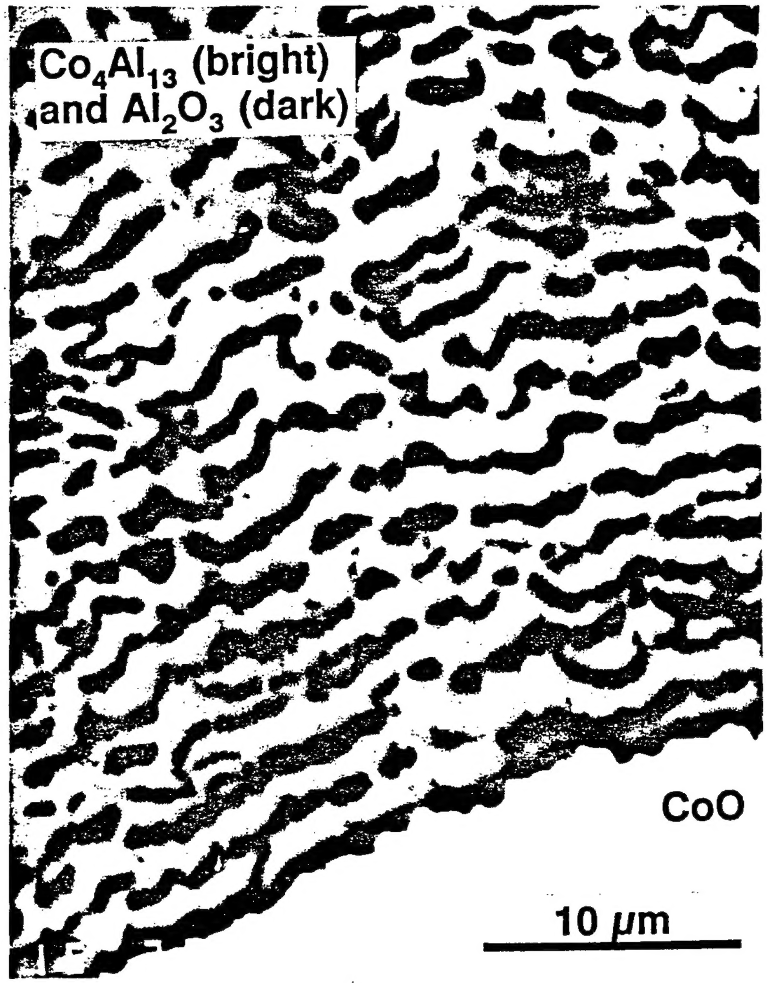

Figure 1: Microstructure observed after reaction between CoO and a cobalt-aluminide intermetallic reactant with 19 at% Co at 1000 °C for 4 hours.

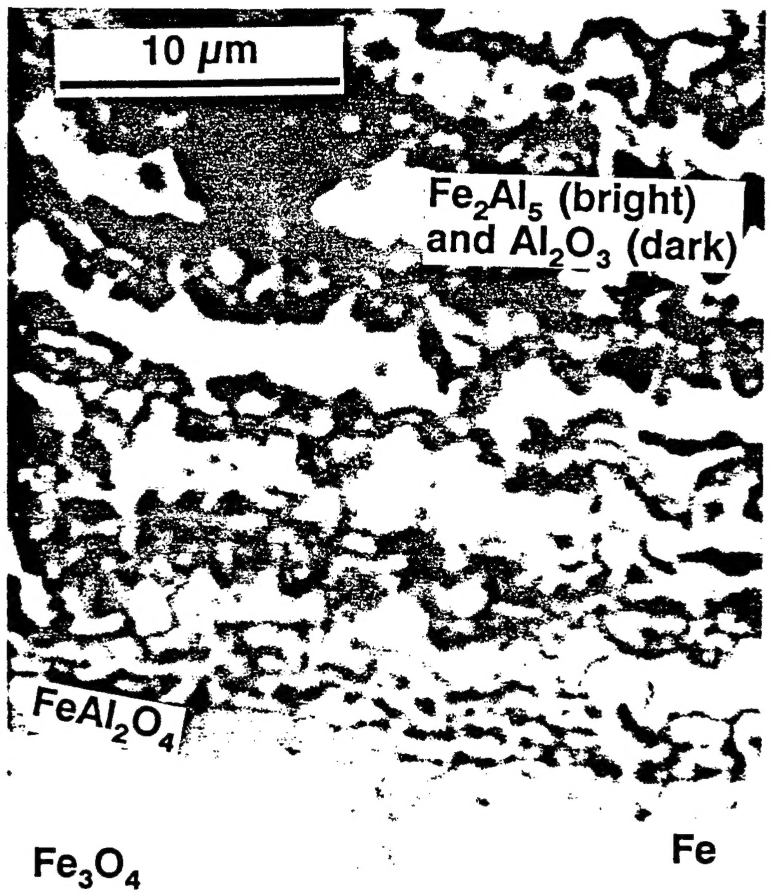

Figure 2: Microstructure observed after reaction between Fe_3O_4 and an iron-aluminide intermetallic reactant containing ~ 28 at% Fe at 1160 °C for 4 hours.

system, we have looked into the reaction between Fe_3O_4 and an iron-aluminide intermetallic reactant containing about 28 at% Fe. The microstructure observed after 4 hours at 1160°C is shown in Figure 2. The layer sequence in the direction away from the Fe_3O_4 reactant is Fe_3O_4, probably FeO (thin layer), Fe (white layer), $FeAl_2O_4$ (thin gray layer), Al_2O_3 (gray), and Fe_2Al_5 (gray), i.e., the periodicity here alternates between Al_2O_3 and Fe_2Al_5. The experimental results discussed above suggest that the formation of periodic layer structures consisting of an oxide and an intermetallic phase is not a unique feature for the reaction between NiO and Al-Ni intermetallic reactants but that such reactions occur also in other Al-containing reaction systems.

As reported earlier by Dieckmann in situ Ni-Al_2O_3 composites have been produced by internal reduction of polycrystalline $NiAl_2O_4$. More recently, in order to understand the basic processes occurring during the partial reduction, $NiAl_2O_4$ single crystals, some containing additions of ZrO_2, were grown and subjected to heat treatments at different reducing conditions. The floating zone technique employing an image furnace was used to grow $NiAl_2O_4$ single crystals. Reduction experiments using single crystalline undoped and ZrO2-doped $NiAl_2O_4$ samples were performed. The microstructures obtained were analyzed with regard to features like pore formation, cracking and the role of the ZrO_2 dopant.

Dieckmann had also tried to perform partial reduction experiments with a load applied to a sample being reduced in such a way that one side of the sample is under tension while the opposite side is under compression. The goal of these experiments was to find out whether stresses can significantly influence the microstructure obtained by partial reduction. Unfortunately, because the existing equipment did not allow one to carefully control the force acting on the sample during the reduction all samples broke during the experiments. In view of the fact that our grant is running out it was not possible to modify the equipment as needed for getting meaningful results. However, we anticipate to perform this type of experiment sometime in the future with more suitable equipment, provided that sufficient funding becomes available.

The primary goal of Giannelis' sol-gel based effort at producing Ni/alumina cermets has been to synthesize composites of fine and highly dispersed microstuctures and then deduce the mechanical properties of these materials, searching carefully for connections between chemistry, microstructure, and fracture behavior (i.e. fracture toughness and fracture strength). The earliest success of the sol-gel synthesis effort was the production of near-fully dense cermets which possessed novel microstructures ranging from a dispersion of isolated, nanosized Ni particles embedded in an alumina matrix to a highly interconnected network of the Ni and alumina phases, both of which were continuous throughout the composite. This was later followed by thorough mechanical investigation of the cermets including such properties as stiffness, hardness, and fracture toughness. In addition, microstructural analysis techniques were devised which served as highly successful "yardsticks" in comparing the often very different microstructures exhibited not

only by the sol-gel based syntheses but also by the internal reduction and displacement reaction syntheses used by other researchers under the AFOSR grant.

The more recent progress areas fall into three categories. First, the fracture toughness characteristics of the sol-gel derived Ni/alumina materials have been thoroughly documented. These characteristics have essentially consisted of significant R-curve behavior, especially in the composites of 20 vol.% Ni or greater. Furthermore, the results of some chemical doping experiments have shown that the addition of chromium oxide to the alumina matrix can enhance the toughness of the cermets, a hypothesis theoretically proposed by others again working under the AFOSR grant. Second, fracture strength testing has revealed a great deal about the type of microstructure optimal for overall composite fracture behavior. In this regard, while increasing the effective size of the metallic phase is favorable for fracture toughness, this very same microstructural alteration generally also decreases fracture strength. Hence, an intermediately sized microstructure is actually best. Finally, focusing exclusively on fracture toughness, it has been found that coarsening of the metallic phase (to enhance toughness) can only be performed until a certain critical size is obtained beyond which interface debonding between the Ni and alumina phases begins to occur. Of course, due to the large CTE mismatch between Ni and alumina, debonding between these phases should eventually be expected. However, very few previous cermet studies (encompassing numerous metal-ceramic systems) have actually investigated the microstructural size at which this debonding occurs.

Burlitch's chemical synthesis program has sough to: (1) discover and develop novel *in situ* syntheses of Ni-Al_2O_3 composites, especially through the use of displacement reactions; (2) develop new routes to incorporate dopants, especially -Cr_2O_3, to strengthen the metal-ceramic interface in the Ni-Al_2O_3 composites; (3) prepare composites that are easy to model theoretically: (4) chemically and spectroscopically characterize the composites; (5) prepare samples of composites suitable for mechanical testing, and (6) refine the synthesis and sample preparation techniques to optimize the mechanical properties at high temperature.

As described in the 1996 Report, sol-gel reactions were used to prepare nickel-alumina composites. The mechanical properties of the undoped composite are somewhat better than those of pure alumina but are worse than those of the coarser composites prepared from displacement reactions. Nickel-alumina composites with chromia added as a dopant to the alumina had improved mechanical properties, especially when only 7% Cr was added; the fracture toughness of this composite (8-9 MPa·$m^{1/2}$) was higher than any prepared in this project previously. In an effort to increase the fracture toughness even more, Ni-Al_2O_3 composites were prepared from commercially available fibers (RATH Performance Fibers, Alcen Mat) in a three stage process. First, the fibers were coated with a thin layer of chromium oxide, using a CVD process previously described in our lab. Then the fibers were coated with nickel metal using an

electroless process.[2] The target composition was 50% Ni by weight. The doubly coated fibers were hot pressed (under argon at 1400 °C) to give a dense pellet. The physical properties of the new composite are being evaluated by Prof. A. Zehnder and coworkers.

B. Mechanical Testing

Zehnder's work has concentrated on completing the installation of the high temperature testing equipment, analyzing the effect of rising fracture resistance curves on measured toughness and refining the room temperature fracture toughness tests by using 3D finite element modelling and laser interferometric measurement of the crack opening displacement. He has also worked with Burlitch to characterize the mechanical properties of a composite he made by infiltrating alumina fibers with Ni, then consolidating the system under heat and pressure. The fracture toughness, density and elastic moduli were measured, and shown to have high porosity and hence low fracture toughness. The processing temperatures were so high as to result in unwanted reactions. Further work is needed to see if the problems of porosity and reactions can be lessened to the point where the potential of such a material can be evaluated.

C. Micromechanical Modelling

Hui's goal is to develop basic rules and criteria for optimizing the mechanical behavior of in-situ processed metal matrix composites operating under high temperature conditions. Analytic and numerical models are being developed to simulate the deformation and fracture properties of the controlled microstructures obtained by the in-situ processes.

A combined experimental (with Zehnder's group) and computational study is carried out to characterize the interfacial strength of the interfaces between nickel and alumina. Computational cohesive zone (CCZ) model is used to model the deformation behavior of the interface. Experiments were done using a sandwich specimen consisting of a thin nickel plate bonded between two alumina plates. These experiments were simulated on a computer to extract the parameters for the CCZ model. It is shown that the work of rupture of a composite reinforced by nickel platelets, does not vary linearly with the size of the inclusions (platelets) as predicted by many theoretical models. This is due to the partial debonding of the nickel/alumina interface. We show that interfaces which are neither too or too weak contribute most to the fracture toughness of the composite. Effects of various parameters of the CCZ model are investigated and it is shown that the most dominant parameter is the interface strength . Effects of the residual thermal stresses are also investigated and it is shown that these stresses can enhance the fracture toughness by almost 16%.

The chevron notch three point bend test specimen is often used for measuring the fracture of brittle materials such as ceramics. Specimen size are often very restricted when testing

advanced material due to limited volume of material available. Since the minimum chevron notch width is limited by the size of cutting wheels or wire saw used to produce it, the notch width becomes large in relation to the sample for a sufficiently small sample. Using finite element analysis, Hui showed that the notch width has an important effect on the stress intensity of short cracks. The minimum in the normalized stress intensity factor versus crack length is lost, rendering the usual analysis of the experimental results invalid and contributing greatly to decreased fracture stability of such specimens. Previous analytical and numerical studies do not take into account the width of chevron notch. Based on our calculations, a guideline to permissible notch width is recommended.

Together with Phoenix's group, Hui has developed an exact theory for the 'single filament composite test'. These equations differ from those formulated by others who have made a priori assumptions on the shape of the fragment distribution that are shown to be incorrect. Furthermore, we have obtained explicit closed form solution of the governing equations for arbitrary Weibull modulus and for random initial breaks with exponentially distributed spacings of a given normalised rate along the fiber. This closed form solution is applied to the study of the strength of large fiber-reinforced ceramics composites. The ultimate strength of such composites is obtained in closed form. Expressions are given in terms of elementary functions which allow computation of the composite ultimate strength to any degree of accuracy. Our results show that the composite strength is unique and occurs at a finite stress for all Weibull modulus, contrary to assertions made by others.

Phoenix is developing computational, micromechanical models for stochastic failure in ceramic/metal composites. The models and capture the random initiation, growth and coalescence of local damage to produce catastrophic cracks. In the model, interfaces may slide according to a viscous law or may crack. In one unified framework he is interested in the effects of randomness in both the microstructural geometry and the local constitutive behavior (flaws, void growth) on (i) the overall strength statistics for material specimens including size effects, (ii) fracture parameters for specimens having an existing crack (statistical fracture toughness, R-curve behavior, energy release rates) and (iii) creep rates and creep-rupture behavior. One goal is to develop computational techniques for the micromechanics which are two to four orders of magnitude faster than current numerical methods. These methods are to be used in Monte Carlo simulations of large systems (numbers of fibers or grains) involving many hundreds of random replications in order to understand the lower tail behavior and other subtle features of the distributions for strength and lifetime important for scaling to large material volumes. Thus new analytical and numerical techniques are being pursued beyond the usual finite element, boundary element and spring network models. Two microstructures have been studied: Microstructure 1, involving parallel

elastic fibers or elongated grains in a viscous or viscoelastic matrix, and Microstructure 2, involving a hexagonal array of grains with cracks placed along interfaces.

In the case of Microstructure 1, Phoenix (in collaboration with Ph.D. student Irene Beyerlein) has developed an efficient computational technique to analyze the time dependent, stress redistribution in a laminar composite having brittle, elastic fibers with random discontinuities embedded in a matrix which creeps. The matrix response is governed by linear viscosity, with either constant or time dependent, power-law creep constitutive behavior. Recent concepts of break-influence superposition (BIS) in a Hedgepeth, shear-lag framework are modified to obtain the solution for an arbitrary array of pre-existing fractures from the time-dependent solution of stresses for an isolated break. The calculations yield the time evolution of the tensile profiles in the fibers and shear stress profiles in the matrix for various constitutive creep law exponents. We present and compare results for an infinitely large, two-dimensional composite lamina under a remotely applied tensile load and which has a microstructure that is locally or periodically damaged in terms of fiber break patterns in both the transverse and longitudinal directions. The model is a key precursor for analyzing failure of fibrous composites where fibers may initially have random discontinuities and may fail sequentially in time at random flaws due to shifting overload profiles near existing breaks. The end result would be catastrophic fracture resulting from eventual localization depending on the model parameters. Progressive cavitation and growth of microcracks in silicon-nitride ceramics with a glassy interphase is an example of a problem that can be analyzed by the present approach.

For Microstructure 2, Phoenix (in collaboration with Ph.D. student Ken Burton) has developed a new and efficient computational scheme, which is a weighted superposition method for calculating stress and displacement fields around an array of kinked and branched cracks made up of straight line segments and existing in a linearly elastic solid loaded in plane stress or plane strain. The method can solve problems of finite domain such as a polygon-shaped plate with prescribed, nonuniform tractions around the boundary. The solution method begins with a crack opening displacement formulation of the exact system of integral equations. The key idea is to use weighted superposition in terms of basis functions arising from certain opening displacement (dislocation density) profiles over straight material cuts, which we call 'cracklets'. The basis functions are chosen to be treatable analytically, producing closed-form solutions (or arbitrarily good closed-form approximations) for their induced stress fields. In addition, they are naturally generated by the local crack geometry in that they capture the natural exponents of important stress singularities associated with wedges at crack kinks or branches. Taken alone, these 'building block' dislocation density profiles are often non-physical and may produce non-physical stress singularities in the basis functions, but after superposition of a sufficient number, non-physical singularities cancel out and the net tractions obtained along crack faces become very smooth. The

weighting coefficients in the superposition are calculated from a least squares fit of the net tractions to the desired tractions (related to the applied loading) at many points along the crack faces. Appropriate constraints are applied to ensure automatically that no non-physical singularities remain. Accuracy is assessed through calculation of the mean square error in the net tractions relative to the desired tractions and a desired level of accuracy is achieved by increasing the degrees of freedom, that is, the number of basis functions used. In the end one has determined the exact solution to a crack problem with net crack tractions that are smooth, bounded and very close to the desired tractions as prescribed by the original boundary conditions. The method has been applied successfully to several benchmark problems including cracks with one and two kinks and a star-shaped crack with several equal arms.

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A. Researchers working on the contract/grant.

Faculty:

Sass, S.L.,	Professor, Materials Science & Engineering,	1.0 month support
Phoenix, S.L.,	Professor, Theoretical Applied Mechanic,	1.0 month support
Burlitch, J.,	Professor, Department of Chemistry,	0.5 month support
Dieckmann, R.,	Professor, Materials Science & Engineering,	0.5 month support
Giannelis, E.P.,	Professor, Materials Science & Engineering,	0.5 month support
Hui, C.H.,	Professor, Materials Science & Engineering,	0.5 month support
Nichols, C.S.,	Professor, Materials Science & Engineering,	0.5 month support
Zehnder, A.,	Professor, Theoretical Applied Mechanic,	0.5 month support

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Kleinsmith, A.L.,	Undergraduate student, Materials Science & Engineering
Reeves, S.Y.,	Undergraduate student, Materials Science & Engineering
Tang, J.,	Undergraduate student, Materials Science & Engineering

B. Articles in peer-reviewed publications, journals, book chapter, and editorships of books.

Materials Letters
In situ formation of composites of alumina with nickel and with nickel aluminide
Jones, Steven A. and Burlitch, James M.
Vol 19, pp 233-235, May 1994

A. Researchers working on the contract/grant for period 4/93 to 8/97

Faculty:

Sass, S.L.,	Professor, Materials Science & Engineering,	1.0 month support
Phoenix, S.L.,	Professor, Theoretical Applied Mechanic,	1.0 month support
Burlitch, J.,	Professor, Department of Chemistry,	0.5 month support
Dieckmann, R.,	Professor, Materials Science & Engineering,	0.5 month support
Giannelis, E.P.,	Professor, Materials Science & Engineering,	0.5 month support
Hui, C.H.,	Professor, Materials Science & Engineering,	0.5 month support
Nichols, C.S.,	Professor, Materials Science & Engineering,	0.5 month support
Zehnder, A.,	Professor, Theoretical Applied Mechanic,	0.5 month support

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Kolhe, R.,	Non-US citizen
Rodeghiero, E	US citizen
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Rodeghiero, E.,	US citizen (ARO Fellowship)

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4) Ishii, S.F., Undergraduate student, Theoretical Applied Mechanic
5) Kim, S.Y., Undergraduate student, Materials Science & Engineering
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